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This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

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Additional inventors are being named on the _____ separately numbered sheets attached hereto					
TITLE OF THE INVENTION (500 characters max)					
LAPPING COMPOSITION AND METHOD USING SAME					
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Respectfully submitted

[Page 1 of 2]

Date February 4, 2004

SIGNATURE

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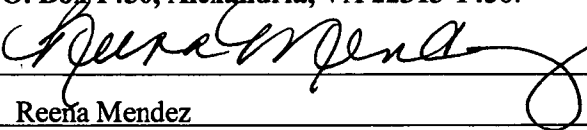
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## **LAPPING COMPOSITION AND METHOD USING SAME**

Lapping, grinding, cutting and polishing (described collectively henceforth as machining) are key manufacturing technologies for shaping various ceramic and metallic materials. A number of factors influence the quality of surfaces shaped by these methods including the size and composition of the particular abrasive grit employed as well as the amount of pressure applied to the surface. Fluids are often also cascaded upon the part during these operations. Among other functions, such fluids remove heat during machining. In addition, such fluids lubricate the abrasive / surface contact area. Such fluids also remove swarf from the part.

10       Rapid swarf removal is desirable in order to efficiently shape the part as well as reproducibly produce smooth, high quality surfaces. Hence it is desirable that the machining fluid exhibits a significant affinity for the surface of the material being shaped. In certain embodiments, such an affinity includes chemisorption upon the ceramic or metallic material; such that its swarf is wetted, dispersed within, and ultimately washed  
15       away by the cascading machining fluid.

As an example, the manufacture of read / write heads for computer hard disks, and the manufacture of the hard disks, includes one or more lapping processes. As those skilled in the art will appreciate, such computer hard disks comprise rotatable disks comprising one or more magnetic materials. Information is written to, and read from,  
20       such magnetic disks using a read/write head.

As those skilled in the art will appreciate, computer hard disks are rotatably disposed in a disk drive unit such that the magnetic disk rotates rapidly as information is

written thereto and/or read therefrom. In order to prevent the read/write head from damaging the disk surface when that disk is rapidly rotating, it is critical that the read/write head be planarized so that it exhibits minimal surface roughness.

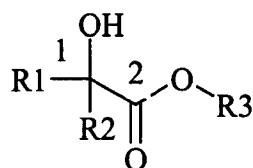
There is an on-going need to maximize the storage density in such computer hard  
5 disks. In order to increase that storage density, the fly height between the rotating disk and the read write head is continually decreasing. As a result, increased demands are being placed upon surface quality of both the read – write head and the hard disk.

One such read/write head is often referred to as a Giant Magnetoresistive (GMR) Head. Such GMR Heads comprise a metallized ceramic material. GMR Heads typically  
10 comprise TiC – Alumina ceramic, sometimes called “AlTiC,” that has been metallized with a variety of ferrous and non-ferrous alloy layers. Prior art GMR Head lapping methods utilize fluids comprising a diamond abrasive dispersed in a mixture of water, various alcohols, and other additives. In order to prevent corrosion of its GMR Head metal layers which would adversely effect the Head’s electromagnetic properties and  
15 operational performance within the computer hard drive.

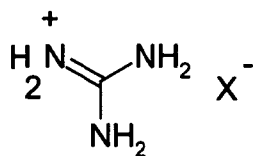
These prior art GMR Head lapping methods are inefficient, exhibiting low AlTiC and metallized layer material removal rates (MRR). A low MRR is undesirable from a manufacturing standpoint because the MRR often comprises a rate-limiting step in the production of read/write heads. In addition, such prior art lapping fluids are highly  
20 alkaline, i.e. pH > 10. Moreover, these prior art lapping compositions are not biodegradable. The alkalinity in combination with the non-biodegradability of these prior art compositions results in significant handling and disposal costs and concerns.

In contrast, Applicants' lapping composition is derived from natural products and are biodegradable. In addition, Applicants' lapping composition poses minimal environmental concerns. Moreover, Applicants' lapping method using Applicants' lapping fluids has a high MRR rate. Applicants' lapping composition is water-based, and includes additives comprising a combination of hydroxyl and carboxylic acid / carboxylate functionalities.

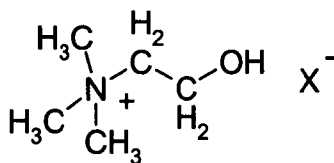
In certain embodiments, Applicants' lapping composition includes salts of alpha hydroxy carboxylic acids (AHC). AHC comprise compounds having structure I, which comprises one or more hydroxyl groups attached to a first carbon atom, where that first carbon atom is covalently bonded to a second carbon atom comprising a carboxyl group.



In certain embodiments, Applicants' lapping composition includes one or more of amine and / or alkali neutralized salts of citric, glycolic, and / or tartaric acid. In these citric acid embodiments, R1 is CH<sub>2</sub>-CO<sub>2</sub>-R4, and R2 is CH<sub>2</sub> - CO<sub>2</sub>-R5. In these glycolic acid embodiments, R1 is methyl and R2 is hydrogen. In these tartaric acid embodiments, R1 is hydrogen and R2 is C(H)(OH)-CO<sub>2</sub>-R6. In these embodiments, R3, R4, R5, and R6, are each independently selected from the group consisting of hydrogen, an ammonium salt, such as tetramethylammonium hydroxide, an alkali metal ion, and alkaline earth metal ion, a guanidinium cation IV, a choline cation V, and the like.



IV



V

5

In certain embodiments, Applicant has discovered that lapping compositions comprising Citric acid show enhanced lapping MRR performance. Table I, below, summarizes MRR using Citric acid based lapping compositions. Lapping was conducted using a Crane Lapmaster Model 15 lapping machine comprising a 15 inch diameter diamond lapping film adhered to the lapping platen.

10



**Table I.**

**MRR Lapping Performance of Various Citrate Salts**

<b>Formulation</b>	<b>Component</b>	<b>Solution pH</b>	<b>Avg. MRR ( g / min.)</b>	<b>MRR Relative to CONTROL</b>
A	DGA Citrate Tribasic	8.94	0.00121	303%
B	DGA Citrate Dibasic	5.06	0.00138	345%
C	MEA Citrate Tribasic	8.5	0.00135	338%
D	MEA Citrate Dibasic	5.23		
E	MEA Citrate Tribasic / Na Polyaspartate Blend	8.8	0.00122	305%
F	Na Polyaspartate	8.1	0.00095	238%
G	Na Citrate Tribasic	8.2	0.00142	355%
H	TEA Citrate Tribasic	6.8	0.00101	253%
I	TEA Citrate Tribasic / Na Polyaspartate Blend	5.05	0.00127	318%
J	TMAH Citrate Dibasic	5.19	0.00137	343%
K	MEA Citrate Tribasic Poly Sodium Aspartate – co- diglycol Aspartamide	8.8	0.00147	368%
CONTROL	Control Conventional GMR Head Lapping Fluid	10	0.00040	--

“DGA” comprises Diglycolamine. “MEA” comprises monoethanolamine. “TEA”

comprises triethanolamine. “TMAH” comprises Tetramethylammonium Hydroxide. Na

Polyaspartate was Baypure DS 100 product supplied by Bayer Corp. (Pittsburgh, PA) having approximately 1500 g/mol. molecular weight

Poly Sodium Aspartate –co- diglycol Aspartamide was prepared via aminolysis ring opening reaction of succinimide repeat units within 3000 MW Polysuccinimide (PSI) ( Bayer Polymers Pittsburgh, PA) dissolved within anhydrous DMSO solvent. Solutions having < 30 wt. % 3000 MW PSI solute are desirable due to apparent PSI solubility & solution viscosity limitations. Diglycolamine was added dropwise to the PSI / DMSO solution at room temperature accompanied by rapid stirring to produce a dark red colored solution. Suitable lapping fluid aspartamide copolymers should be prepared via reaction of between 0 and 75 mol. % of PSI succinimide repeat units with diglycolamine. The remaining, unreacted SI repeat units in the aspartamide copolymer are hydrolyzed via reaction with aqueous NaOH solution in 1;1 molar stoichiometry producing the desired Poly Sodium Aspartate – co- Diglycol Aspartamide copolymer.

Formulations A through K, inclusive, and the Control, had identical Citrate Molality within Distilled H<sub>2</sub>O, namely 0.214 Moles Citrate / Kg H<sub>2</sub>O solvent. Formulations A through K, inclusive, were prepared via dissolution of anhydrous citric acid (A.C.S. Reagent) in distilled water followed by dropwise addition of amines accompanied by rapid stirring until a product having desired stoichiometry was obtained. Because TMAH reagent was used in its pentahydrate crystalline salt form, this material was first dissolved in distilled water prior to its addition to aqueous citric acid solution. Sodium citrate tribasic (A.C.S. Reagent) was purchased from Aldrich Chemical Company ( Milwaukee, WI).

As those skilled in the art will appreciate, dissolution of anhydrous citric acid was highly endothermic, producing a solution having a temperature below about 25 oC, i.e. room temperature. Such an endothermic dissolution is advantageous because the chilled solution produced compensated for the exotherm produced from the subsequent citric acid neutralization.

As those skilled in the art will appreciate, the pKa values for the various amines used follows the order of:

TMAH >> Diglycolamine  $\approx$  Monoethanolamine >> Triethanolamine

During lapping, AHC compounds are believed to strongly chemisorb onto the AlTiC GMR Head specimen surfaces. As the ceramic material becomes abraded, the adsorbed AHC compounds impart a significant anionic charge to the swarf particles. Significant interparticle electrostatic repulsion facilitates rapid removal of the swarf from the specimen during lapping.

In order to minimize corrosion of the GMR Head metallic layers during lapping, it is desirable that the lapping fluid have an alkaline pH. Under alkaline conditions the surface of AlTiC acquires a net negative charge which would normally inhibit the adsorption of these anionic carboxylate compounds. The presence of hydroxyl groups on the citrate anion, however, promotes adsorption even under alkaline conditions. Such adsorption may result from the formation of highly stable cyclic ligands bound to surface metal cations or via strong hydrogen bonding interactions between the hydroxyl groups and the ceramic surfaces.

Formulations F and K showed enhanced MRRs during AlTiC Head Lapping. These rate enhancements likely result, in part, from the combination of pendent carboxylate and amide functional groups in the respective polymeric materials. The amide groups may promote polymer adsorption on the AlTiC surfaces via hydrogen  
5 bonding interactions.

A synergistic MRR effect is observed when lapping AlTiC with a Formulations E, I, and K. These Formulations include both citrate and polyaspartate species. Higher MRRs are observed by using formulations comprising a combination of these components compared to fluids containing either individual component alone. Applicant  
10 has discovered that preferred weight ratios of the polyaspartic (or aspartamide copolymer) to citrate salt range from 0.0 to about 1. In certain embodiments, Applicants' lapping formulation comprises a weight ratios of the polyaspartic (or aspartamide copolymer) to citrate salt range from about 0.1 to about 0.5.

Tables II and III recites Formulations L and M, respectively, comprising  
15 polyaspartate or aspartamide copolymer, in combination with a citrate blend.

**Table II**

**FORMULATION L**

**Polyaspartamide Copolymer / Citrate Lapping Fluid Composition**

<b>Component</b>	<b>Concentration (Wt. %)</b>
Distilled Water	90.7
MEA Citrate Tribasic	7.3
Poly Sodium Aspartate –co- diglycol Aspartamide ( 33 mol. % polymer repeat units amidated)	2.0

5

**Table III**

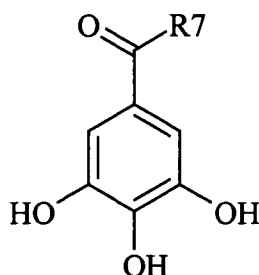
**FORMULATION M**

**Polyaspartate / Citrate Lapping Fluid Composition**

<b>Component</b>	<b>Concentration (Wt. %)</b>
Distilled Water	69.43
Triethanolamine Citrate Tribasic	25.9
Sodium Polyaspartate (Baypure DS 100)	4.67

Applicants' lapping composition further includes lapping fluid formulation  
10 derived from hydroxyl substituted benzoic acids. In certain embodiments, Applicants' lapping composition is formed using Gallic Acid, Compound II where R7 is OH. In certain embodiments, Applicants' lapping composition includes Compound II wherein

R7 is selected from the group consisting of OH, O-R16, N(R17)(R18), and the like, wherein R16 is selected from alkyl, alkali metal cation, alkaline earth cation, cation IV, cation V, and the like, and wherein R17 and R18 are selected from alkyl, aryl, phenyl, and the like.



II

5

In certain of these embodiments, gallic acid is dispersed in distilled water at concentrations similar to those listed in Table I. Due to the highly acidic nature of Gallic acid, Applicant neutralizes the aqueous gallic acid solution using a variety of amines, alkali hydroxides, choline derivatives, guanidine derivatives, or TMAH prior to its use in lapping formulations. Suitable bases include but are not limited to alkanolamines, alkylamines, choline, guanidine derivatives, and the like. Alkaline lapping solutions are desirable to minimize corrosion which may occur during lapping of metallic surfaces. The reaction stoichiometry required to neutralize Applicant's gallic acid embodiments is a function of the pKa of the particular base used.

15

As a general matter, at least one equivalent of base is used for each equivalent of Gallic acid. As a further general matter, Applicant has found it desirable to have lapping fluid solutions comprising a pH greater than 8. As those skilled in the art will appreciate,

such a resultant pH requires excess equivalents of base with respect to the gallic acid used.

For example, a 0.216 M aqueous solution of gallic acid was prepared having a pH of 3.05. Addition of 0.0429 mol. of ethanolamine to 200 ml of this solution

- 5 corresponding to a 1:1 molar acid / base raised the pH to 6.89. Further ethanolamine was added to give a 1.18 amine / acid molar ratio. This formulation had a clear green color with a pH of 8.72. Table IV lists the composition of this Formulation N.

**Table IV**

**FORMULATION N**

10

**Gallic Acid Salt Lapping Fluid Composition**

<b>Component</b>	<b>Concentration (Wt.%)</b>
Distilled Water	94.0
Ethanolamine	2.1
Gallic Acid	3.9

AlTiC bars were lapped using the Formulations recited in Table V under the same conditions as detailed in Table I above.

**Table V**

**MRR Lapping Performance of Various Gallate-Derived Salts**

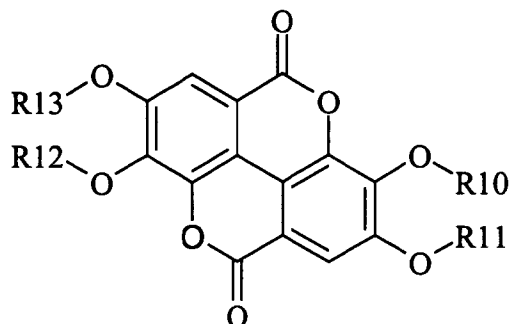
<b>Formulation</b>	<b>Component</b>	<b>Solution pH</b>	<b>Avg. MRR ( g / min.)</b>	<b>MRR Relative to CONTROL</b>
O	MEA Gallate	8.72	0.00093	233%
P	Potassium Gallate (1:1 mole ratio KOH/Gallic Acid)	8.98	0.00110	275%
Q	Potassium Gallate (3:1 mole ratio KOH/Gallic Acid)	10.1	0.00154	385%
R	Potassium Gallate (3:1 mole ratio KOH/Gallic Acid) AGED 48 HOURS	9.1	0.00181	453%
S	Potassium Gallate (Freshly Prepared)	9.1	0.00144	360%

Formulation R is formed using the same starting materials as Formulation Q.

Formulation R is aged, however, for 24 hours before use. During that 24 hour period, the color of the solution changes from a clear, light green color to a very dark green color. In addition, the pH of Formulation R is less than the pH of Formulation Q.

It is known in the chemical arts that Gallic acid dimerizes under aqueous alkaline conditions to form Ellagic acid derivatives, Compound III, where R10, R11, R12, and R13, are selected from the group consisting of ammonium salts, alkali metal ions, alkaline earth ions, guanidinium ions, choline cations, and the like.





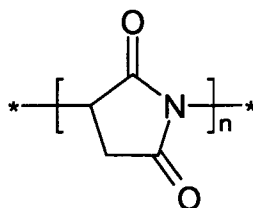
III

Gallic acid derived Formulations O through S are useful because the aromatic hydroxyls are deprotonated at alkaline pH and strongly adsorb upon the alumina surface.

Applicant has found that citric acid derived formulations appear to be less strongly  
 5 adsorbed at alkaline pH values compared to Gallic acid embodiments O through S.

Gallic acid, Ellagic acid, and related polyphenolic derivatives, exhibit low toxicity, and can be readily obtained via the hydrolysis of tannic acid, a natural product derived from wood by products. As those skilled in the art will appreciate, Tannic acid is essentially composed of a mixture of polysaccharides having a high concentration of  
 10 gallic acid esterified along the saccharide polymer chains.

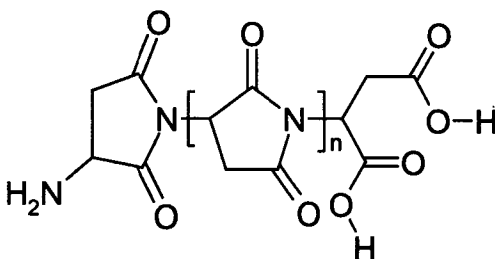
In certain embodiments, Applicants' lapping composition comprises polymeric materials which include a plurality of pendent gallamide moieties. In certain embodiments, these polymeric materials are formed by reacting a polymer which includes a plurality of polysuccinimide repeat units VI with an amino-terminated  
 15 gallamide.



VI

In certain embodiments, Applicant's polymeric material which includes a plurality of gallamide moieties is formed from polysuccinimide XI. In certain embodiments,

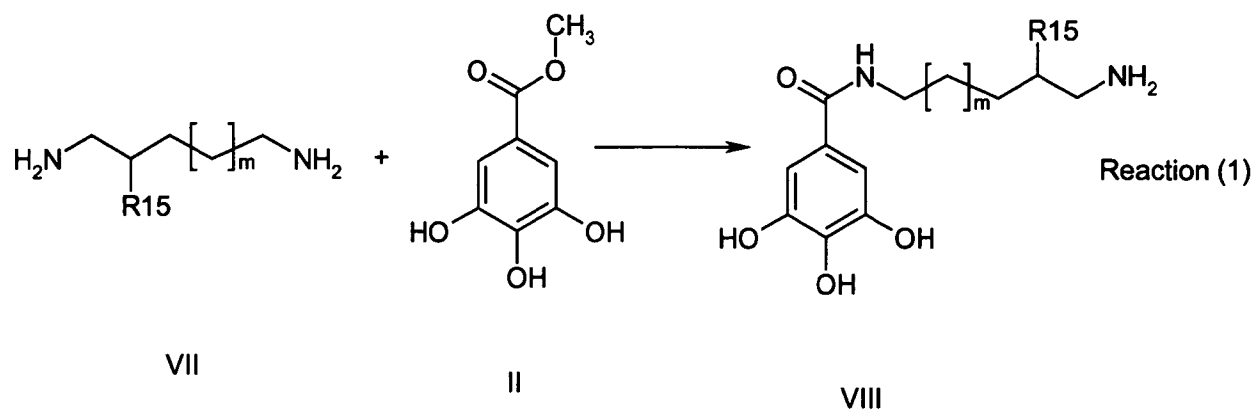
5 polysuccinimide XI has a number average molecular weight of about 3,000 Daltons.



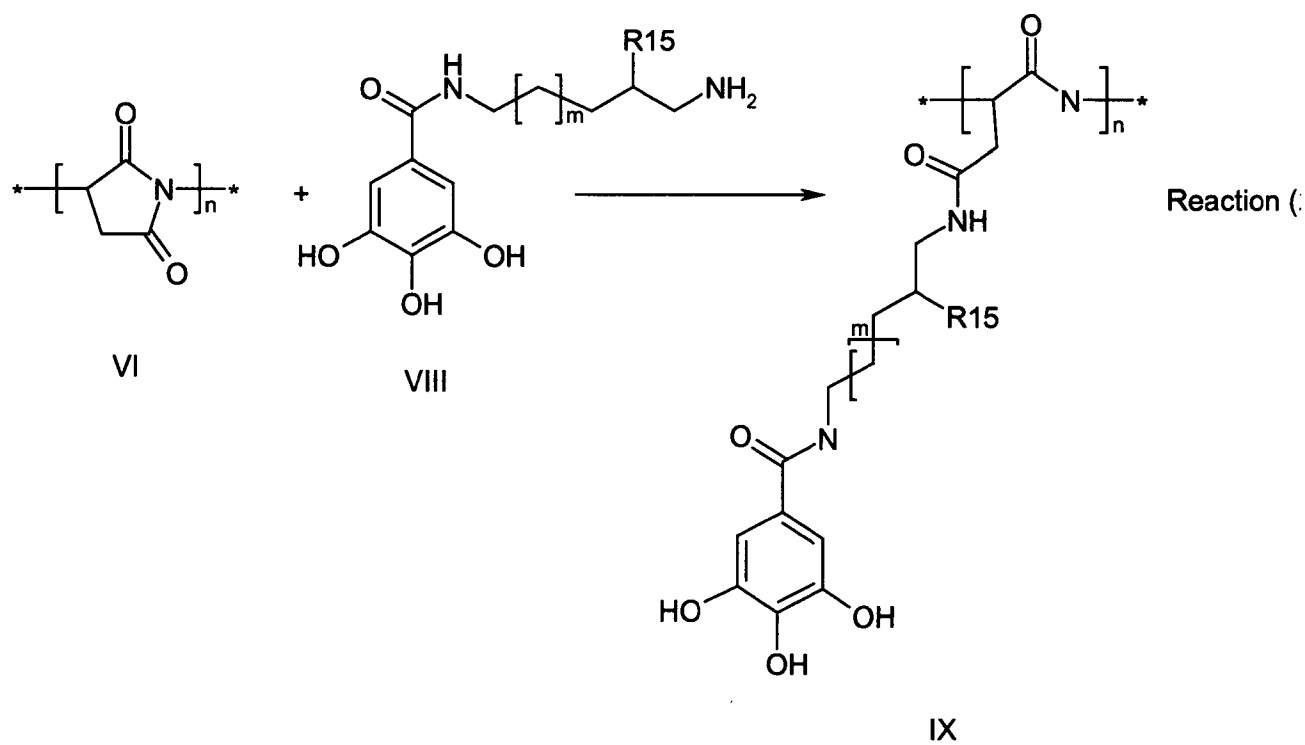
XI

In one embodiment, for example, methyl gallate, Compound II wherein R7 is O-Me, is reacted with diamine VII to give amino-terminated gallamide VIII wherein R15 is

10 selected from the group consisting of hydrogen, alkyl, oxyalkyl, aryl, phenyl, and oxyphenyl, and wherein (m) is greater than or equal to 1 and less than or equal to about 10.



Amino-terminated gallamide VIII is then reacted with polysuccinimide VI to form derivatized polyaspartamide IX



EXAMPLE 1  
Synthesis of Compound VIII

About 6.60 g ( 0.036 mole) of Methyl-3,4,5-trihydroxybenzoate was dissolved in about 46.4 g of Dimethyl Sulfoxide (DMSO) by gently heating the mixture at 45 °C for about 20 minutes with stirring. As those skilled in the art will appreciate, DMSO is a polar, aprotic solvent having low toxicity and good biodegradability. Other polar, aprotic solvents may be used in place of, or in combination with, DMSO. Such polar, aprotic solvents include, for example, N- Methyl Pyrrolidone, Sulfolane, Tetrahydrofuran, or Dimethyl Sulphone.

10        The resulting DMSO solution was then added to a 250 ml round bottom flask ... which had been previously flushed with inert nitrogen gas. About 10.368 g ( 0.08922 mole) of 2-methyl-1,5-pentanediamine, Compound VII, wherein R15 is methyl and (m) is 1, is sold in commerce by duPont under the tradename Dytex A, were then added to that DMSO solution, and the resulting reaction mixture was heated under vacuum at about 15    160 °C for about 8 hours. Diamine 2-methyl-1,5-pentanediamine was selected on the basis of its nucleophilicity combined with its high  $pK_{a1}$  value of 11.2.

During the reaction, methanol was removed from the reaction mixture as it was formed as the product from the aminolysis reaction occurring between the gallate ester and Diamine 2-methyl-1,5-pentanediamine. A clear, slightly reddish colored solution 20    comprising about 26.6 weight percent compound VIII was obtained.

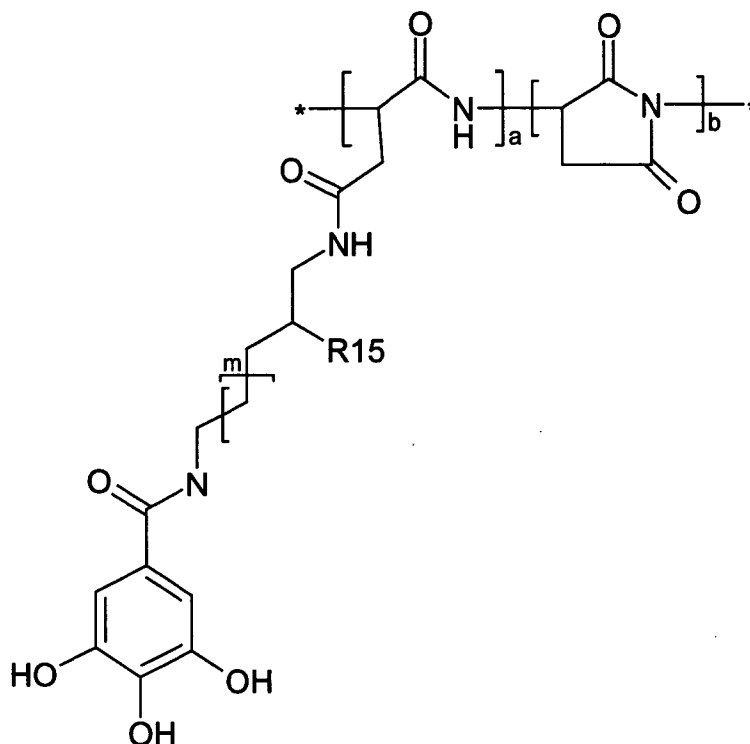
## EXAMPLE 2

### Polyaspartamide IX / X Preparation

About 7.82 g of 3,000 MW Polysuccinimide, polymer VI, sold in commerce by Bayer Chemicals Corp. Pittsburgh , PA under the tradename Baypure, was dissolved in  
5 about 33.0 g DMSO at 55 °C. This yielded a solution comprising about 19.1 weight percent polymer VI

About 19.72 g of a DMSO solution of compound VIII, from Example 1, was diluted with about 8.61 g additional DMSO. This DMSO solution was rapidly mixed with the previously prepared DMSO / polymer VI solution.

10 In certain embodiments, a stoichiometric excess of Compound VII was added in an amount such that all the succinimide moieties were ring-opened. In other embodiments, less than a stoichiometric amount of VII was used, thereby giving co-polymer X having both succinimide linkages and substituted aspartamide linkages.



X

For example, in one embodiment about one-fourth of the succinimide repeat units in PSI were ring-opened using Reaction 2 to give co-polymer X wherein a is about 0.25(b). An exotherm was noticed upon mixing accompanied by a darkening of the solution such that it became sanguine in appearance. The solution was allowed to stand at room temperature for 30 minutes resulting in a clear, homogeneous, sanguine colored solution.

### EXAMPLE 3

#### Lapping Solution Formulation

About 196.5 g of a solution comprising about 1.08 weight percent NaOH in water was combined with a DMSO solution of co-polymer X wherein a is about 0.25(b). The reaction mixture was stirred for about 1 hour at room temperature to give a solution

having a pH of about 10.82. Table VI summarizes the composition of the aqueous lapping fluid prepared.

TABLE VI

Component	Concentration (Wt. %)
Sodium Polyaspartic-co-aspartamide copolymer (25% pendant gallamide)	5.91
DMSO	18.5
Distilled Water	75.59

- 5 AlTiC bars were then lapped using the method described about. An average MRR of 0.00133 g/min. was obtained using this lapping composition of Table VI.

The measured MRR for the composition of Table VI is significantly greater than the MRR of 0.00095 g/min. MRR measured when lapping AlTiC using a 25 weight percent solution of Baypure DS 100.

- 10 In other embodiments of Applicants' composition, pendant amine functionalized Gallamide VIII is reacted with other polymers to produce polymeric lapping fluid additive having both electrostatic and steric stabilization to polished swarf particles. Such other polymers include, for example, maleic anhydride copolymers, such as Scripset 1000 – Polystyrene-co-maleic anhydride polymer obtained from Sartomer Company Inc.
- 15 Exton, PA, or Gantrez AN - Polymethylvinylether-co-maleic anhydride obtained from International Specialty Products Inc. Wayne, NJ. In these embodiments, the amine functionalized gallamide VIII is reacted with the maleic anhydride copolymer in a polar

aprotic solvent, followed by dissolution in aqueous alkaline solution to produce a polyelectrolyte lapping fluid additive.

In yet other embodiments, the amine functionalized gallamide VIII is with isocyanate prepolymers, acrylate oligomers, and/or epoxy oligomers, to produce water  
5 dispersible lapping fluid additives.

In other embodiments, polyethyleneimine (PEI) is reacted with methyl gallate to give a polymeric material comprising pendent gallamide moieties. As those skilled in the art will appreciate, PEI has a brush like structure, and is known in the art for its ability to serve as a dispersant in a wide variety of ceramic slurry formulations.

10 Applicant's gallamide functionalized PEI brush polymer is synthesized by adding methyl gallate to PEI either neat, or in a solvent, and allowing that reaction mixture to stand for several days at room temperature. During this reaction period, an aminolysis reaction occurs between the primary amine groups present on PEI and methyl gallate.

In certain embodiments, a stoichiometric excess of methyl gallate is used to react  
15 with essentially all of the amino moieties in the PEI polymer. Heating the reaction mixture in a nitrogen atmosphere at temperatures greater about 67 °C, i.e. the boiling point of methanol, drives this aminolysis reaction to completion. Aqueous solutions of these functionalized PEI polymers typically have pH values greater than about 9, and are suitable lapping fluids.